Art. XIV.—*Account of the Separation and Identification of a Kaolin Incrustation on Pyrolusite from Broken Hill.*

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(Communicated by Professor Orme Masson).

(With Plate XXIV.).

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The specimen investigated was obtained by Professor Orme Masson from Broken Hill. It is bluish white externally, and consists, for the most part, of a lacework of rods and films. Parts of it are more solid and rather botryoidal in appearance. Any part that has been broken shows that the colour is due to a thin white film. Inside this the mineral appears to consist of black pyrolusite, enclosing occasional rounded grains of glassy quartz. No other mineral can be observed macroscopically.

The Main Problem was to Identify the Incrustation or Film.

1. Several sections of the specimens were therefore prepared for microscopic examination. These were cut across the more solid parts of the mineral, and showed that the external layer is transparent and crystalline. No definite crystal shapes can be seen, but the mineral shows weak double refraction. The refractive index is very low. In the sections prepared, this mineral is always in direct contact with the pyrolusite, and even when it occurs very close to the other minerals is always separated from them by a thin film of pyrolusite. From a number of measurements made:—

Greatest thickness	-	-	.083 mm.
Least ,,	-	-	.024 ,,
Mean ,,	-	-	.054 ,,

2. The mineral, after powdering and passing through a fine wire sieve, was treated with hot concentrated hydrochloric acid, and it was found that ultimately everything dissolved except the incrustation and the quartz. Since the incrustation is a silicate, it was necessary, in order to determine its quantitative composition, to separate it from the quartz prior to analysis. The great difficulty experienced in this separation was due to the fact that the specific gravities of the two materials are very close together, being 2.61 and 2.65 respectively, the difference of specific gravity being the particular difference on which the methods of separation depended.

The first attempt at separation was made by use of a diffusion column using Sondstadt's[1] solution, of specific gravity 3.04, at the bottom of a tube covered by a diluted Sondstadt's solution of specific gravity slightly less than 2.6. Many modifications of the method were tried, but all failed to bring about any separation, as shown by examination of minute samples under the microscope.

The next method attempted was to shake up the mixed powder with Sondstadt's solution in a test tube and allow it to stand. Both minerals then rose to the surface. A single drop of water was then added, and the process repeated until at last a point was reached when the powder was divided into two layers, one of which sank and the other came to the surface. Samples of each layer were taken, and on examination each proved to be a mixture of the two minerals practically identical with the original mixture, both as to size of particles and as to proportionate quantities present. Several trials of this method always gave the same result.

The next step was to put this same solution with the suspended powder in the centrifuge. Two layers were obtained, the top one of which was poorer and the bottom one richer in quartz than the original mixture. The top layer was now stirred up without disturbing the bottom layer, and on again centrifuging more quartz left the top one. On repeating the operation three or four times the top layer was obtained quite

1 Sondstadt's solution. An extremely concentrated solution of Mercuric Iodide in aqueous Potassium Iodide solution.

free from quartz. To bring about a perfect separation it was found advisable to wash away from the powder the constituents of which are to be separated all the very fine particles so as to leave the grains about one size.

The result of the above experiments is to show that a separation of two constituents of the powder having specific gravities within .04 of each other, and having particles of maximum diameter .015 mm. is impossible by means of Sondstadt's solution useless the centrifuge be used as above described.

3. The isolated incrustation was examined. An analysis gave the following results :—

	Isolated Incrustation.	Calculated from formula $Al_2O_3\ 2H_2O\ 2SiO_2$	Analysis of Kaolinite given in Dana, quoted from Tookey & Dick, Percy's Metallurgy.
Al_2O_3	46.92	46.5	46.53
H_2O	14.09	14.0	13.87
SiO_2	38.99	39.5	38.93
	100.00	100.0	99.33

We see that the approximation of the composition of the material to that represented by the formula $Al_2O_3 . 2H_2O . 2SiO_2$ is very close.

The specific gravity was determined approximately in the following manner:—Some Sondstadt's solution was diluted down until it was seen that particles of the incrustation just floated. The specific gravity of the liquid was found to be 2.61 by Sprengel tube. Hence we may call this the specific gravity of the kaolin.

The account of the examination of the optical properties of the incrustation has already been given.

4. It was found, as a result of this investigation, that this incrustation is kaolinite.

Its percentage composition, specific gravity and optical properties under the microscope, together with the property of retaining its combined water at 150 deg. C., though parting with it at a red heat, agree with those given in Dana for kaolinite. Further, its properties under the microscope correspond with those of a known specimen of kaolinite mounted as a microscope slide for comparison.

Notes on the Remainder of the Mineral.

1. Microscopic Investigation.—Included in the pyrolusite is a colourless mineral generally occuring in aggregates of small crystals. Occasionally a single crystal section can be found. These sections are usually hexagonal in shape. One was isotropic, and the angle between its sides was 120 deg., so that the mineral would appear to belong to the hexagonal system. The crystals are too small to give a satisfactory interference figure with convergent polarised light. The refractive index is high, and the interference colours yellows and greys.

There is a third mineral present in minute quantities which can be easily picked out by its red colour. It gives no very satisfactory results in the slide, but can be obtained as separate crystals by powdering some of the original specimen, damping the powder, and then adding water. The red mineral is found floating on the surface. It is evidently not wetted by the water, and remains floating on account of the surface tension. It may also be separated on account of being the last mineral to dissolve on treating the powdered sample with concentrated hydrochloric acid. Examined under the microscope, it is seen to occur as well formed minute tabular crystals bounded by four rectangular sides. Some crystals are pleochroic and doubly refracting, and give a straight extinction. Others which are not isotropic remain dark between the crossed nicols, and give a uniaxial interference figure. The mineral is therefore tetragonal. It may be wulfenite ($PbMoO_4$).

Quartz also occurs throughout the pyrolusite in rounded grains of considerable size, which contain many liquid inclusions.

2. Chemical Investigation.—A bulk analysis of the specimen gave the following result:—

Insoluble in concentrated hydrochloric acid	Kaolin - -	4.14
	Quartz - -	5.22
Soluble in concentrated hydrochloric acid	MnO_2 - -	43.00
	PbO - -	25.96
	$Al_2O_3 + Fe_2O_3$ -	20.76
	Sb_2O_5 - -	.30
	Cu - - -	Trace
		99.38

Fig. 1.

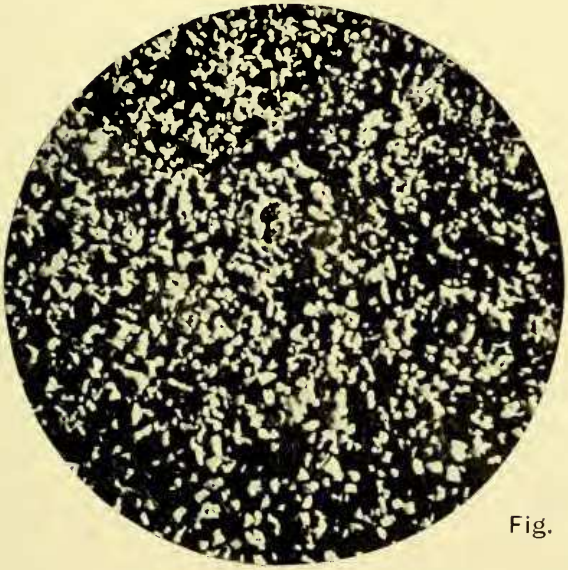

Fig. 2.